http://dx.doi.org/10.4314/jpb.v8i2.11
Vol. 8 no. 2, pp. 135-140 (September 2011)
http://ajol.info/index.php/jpb

Journal of
PHARMACY AND BIORESOURCES

Effect of some stabilizing agents on globule characteristics and rheological properties of oil in water emulsions

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Received 28th June 2011; Accepted 29th July 2011

Abstract

This study investigated the effects of some stabilizing agents (cassava, maize and bentonite mucilages) on globule characteristics and rheological properties of oil in water emulsions. Emulsions were prepared by mixing varying proportions of the mucilages with Arachis oil in the ratio of 60:40 (oil: water) with the aid of a mixer. Parameters evaluated were globule sizes and numbers, and flow properties of fresh and aged emulsions. Generally, starch mucilages and their emulsified systems displayed higher viscosities compared with that of bentonite mucilage and its emulsified system. For instance, mean values of viscosities of the mucilages at 4%w/v expressed in centipoises (cP) were 1448 ± 1.2 (cassava mucilage), 1140 ± 0.4 (maize mucilage) and 36 ± 2.1 (bentonite mucilage), while the emulsified systems were 1730 ± 1.3 (cassava emulsion), 1206 ± 0.8 (maize emulsion) and 22 ± 0.9 (bentonite emulsion) respectively. Globules were uniformly distributed. There was a general increase in globule sizes with time ranging from 12.44 – 23.16 μm (cassava emulsion), 18.92 – 31.12 μm (maize emulsion) and 26.17 – 33μm (bentonite emulsion) respectively and this is an indication of globule coalescence. However, the globules ruptured and disappeared within two weeks of storage. Globule coalescence was inversely related to the viscosity of the dispersed medium. The study has shown that inclusion of viscosity impacting agents in the dispersion medium in emulsified systems play vital role in the stability of emulsions. This finding has useful application in the formulation of drugs in dispersed systems.

Keywords: Viscosity impacting agents; Mucilage, Globule stability, Dispersed medium; Flow

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INTRODUCTION

Emulsions are disperse systems made up of two immiscible phases (oil and water) one of which is finely subdivided and uniformly distributed in the other and the system is stabilized by the presence of an emulsifying agent (Martin 1993). As a pharmaceutical dosage form emulsions have found wide applications in the formulation of hydrophilic and hydrophobic drugs together in the same dosage form, taste masking, prolongation of action, improved stability, and in the separation of incompatible components in the same dosage form amongst others. Emulsions generally are regarded as thermodynamically unstable due to the high interfacial energy existing on the surfaces of the dispersed globules. The ability of the dispersed globules to retain its initial character and remain uniformly distributed

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ISSN 0189-8442

throughout the continuous phase is a measure of its stability (Richards, 1972 and Okor and Obaduni, 1992). Stability of emulsion can be achieved in various ways such as the lowering of the interfacial tension between the two opposing phases, formation of rigid films on globule surfaces mainly by surface active agents and a marked increase on the viscosity of the dispersed medium as a means of reducing the rate of coalescence of dispersed globules. This last approach often makes use of viscosity impacting substances such as gums and mucilage from naturally occurring sources such as carbohydrates and starches e.g. acacia, sodium alginate and tragacanth, semi synthetic and synthetic polymeric substances such as polyvinyl pyrrolidone (PVP), methylcellulose and carboxymethyl cellulose. In all these instances the property exploited is the ability of the viscous mucilage to retard the coalescence of the droplets in the dispersed medium. Mucilages have been employed in ancient times in the formulation of pharmaceutical dosage forms such as binders, film formers, suspending agents and thickeners (Gaiker et al, 2011).

Polymeric substances are usually expensive and require high foreign exchange to import and thus make formulation very expensive. However, carbohydrates and starches are readily available staple foods in the tropics, they are cheap and starch extraction processes are cheap and do not require high technological techniques. Hence the objective of this study therefore was to investigate the effect of some viscosity impacting properties of locally available starches on the globule characteristics and rheological properties of emulsions.

EXPERIMENTAL

Materials: Cassava tubers and maize grains were sourced from a local market in Yenagoa, Bayelsa State of Nigeria, while bentonite was obtained from British Drug House, (BDH) and they were investigated as viscosity impacting agents in the stabilization of emulsions. Magnesium trisilicate powder was obtained from British Drug House, (BDH) and used in this study as a model of diffusible drug. All other chemicals were of reagent grade.

Extraction of starches. The starch was extracted from cassava tubers of a particular specie *Manihot utillissima* and purified by a modified procedure described previously by Eichie and Okor, 2002 as follows: Cassava tubers were peeled, washed and rasped to a pulp using a local blending mill in the community. Starch was extracted from the pulp by overnight immersion in excess water followed by straining with a nylon cloth. The resulting suspension was allowed to stand for 18 hours, the supernatant decanted and washed several times with distilled water. The slurry was strained through a fine muslin bag into a cake and sun dried for 48 hours. The fine powder was finally dried in a hot air oven at 50^0C for 6 hours, (Hillock *et al*., 2002).

On the other hand the maize starch was extracted using the following procedure: The grains of particular specie Zea mays were soaked in 0.2% solution of sulphuric acid to render the grains soft. The soft grains were milled in a blending mill in the community after thorough cleaning. The starch was washed off with excess distilled water using a clean muslin cloth by rubbing. The starch suspension obtained was left to stand for six hours, the supernatant decanted and the resulting starch was shed severally with excess distilled water. The slurry was finally strained of water and the resulting cake was sun dried for 48 hours and finally dried in a hot air oven at 50^oC for 6 hours, (Evans, 2002).

Preparation of mucilage. Varying concentrations 2-5 % w/v of the starches (cassava or maize) was prepared by dispersing the appropriate weights of the starches in 20 ml distilled water at room temperature, 28^0C to form a suspension. The resulting suspension was made up to the final volume of 100 ml by the addition of boiled water at 100^{0}C to form the mucilage. To form the bentonite mucilage, varying concentrations (2-5%w/v) of bentonite powder were dispersed in distilled water for 24 hours with occasional stirring. 0.25 %w/v methylparaben was added to all mucilages for preservation.

Preparation of the emulsions. Three types of emulsions identical in content of oily phase (arachis oil 40 % v/v) were made with cassava and maize starches or bentonite mucilages respectively. To form the emulsions, the oily phase was gradually added to the aqueous phase (60 ml mucilage) while stirring for 5 mins with a mixer (Biomedical Engineering Inc. USA) to form a homogenous dispersion.

Table 1: Rheological properties of the freshly made mucilages and emulsions

Stabilizing agent	Mean viscosities in Centipoise (cP)	
	Mucilage	Emulsion
Cassava	1448 ± 1.2	1730 ±1.3
Maize	1140 ± 0.4	1206 ± 0.8
Bentonite	36 ± 2.1	220 ± 0.9

Table 2: Effect of ageing on the rheological properties of the emulsions

Storage time (days)	Changes in the viscosity, Centipoise (cP)					
	Cassava		Maize		Bentonit	
	Mucilage	Emulsion	Mucilage	Emulsion	Mucilage	Emulsion
1	1448±1.2	1730±1.3	1402±0.4	1706±1.3	36±2.1	220±0.4
2	1391±0.2	1710±0.5	1311±1.6	1634±1.4	30±2.0	190±1.0
3	1306±1.1	1214±2.0	1246±0.4	1141±1.7	22±1.0	177±0.3
4	1228±0.5	1185±0.9	1206±0.3	1100±1.3	17±1.2	161±0.2
5	1180±0.2	1150±0.8	1099±1.1	1075±2.6	10±1.3	156±0.3
6	1101±1.3	1133±0.7	1016±0.4	1040±1.1	6±1.1	111±0.7
7	1034±1.1	1055±0.8	989±0.8	1001±2.2	1.2±0.2	92±0.9

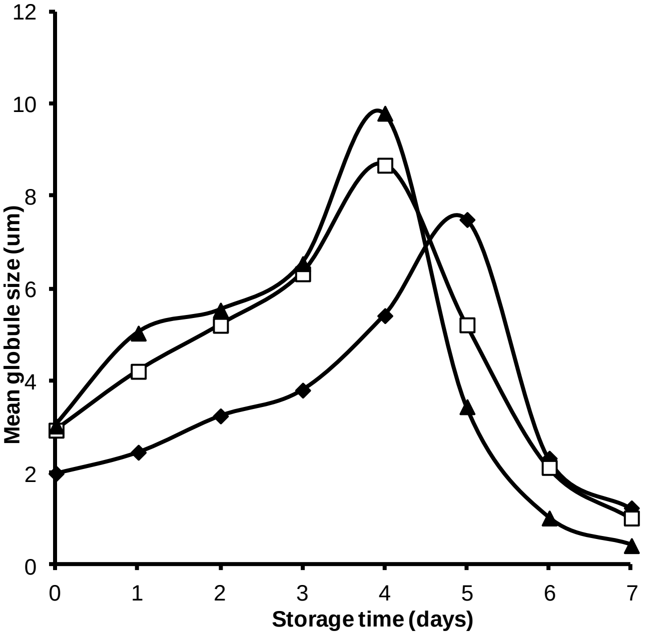

Fig 1: Effect of ageing on mean globule size of emulsion: (■) cassava starch, (□) maize starch and (▲) bentonite emulsions respectively.

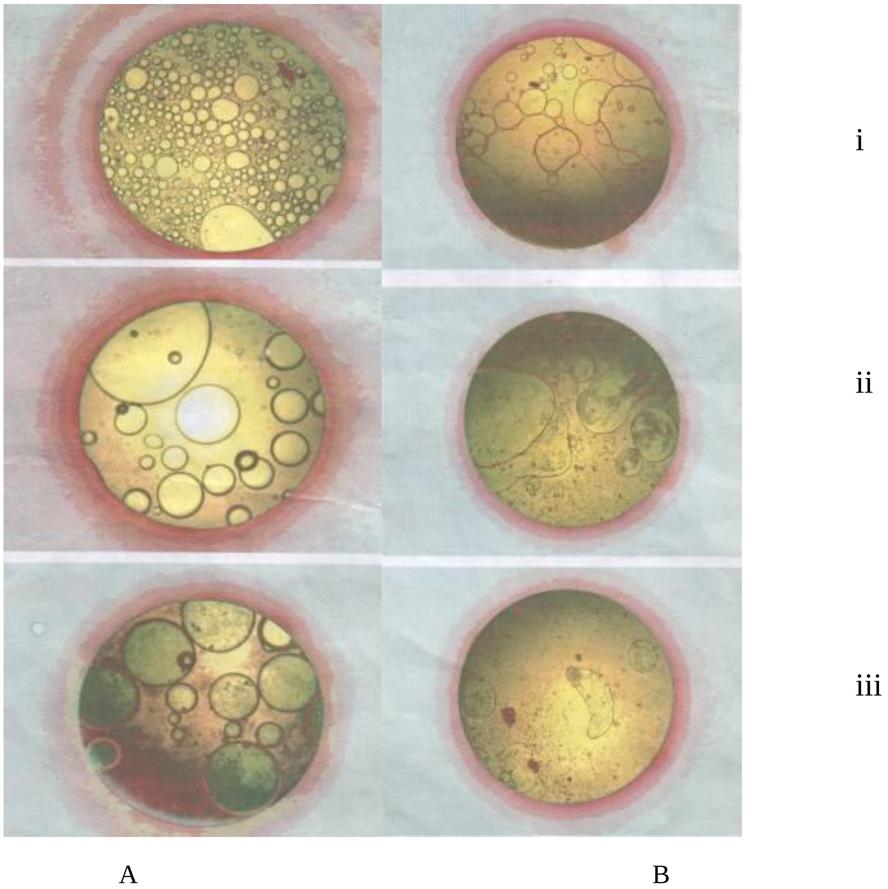

Fig. 2: Photomicrographs of freshly made (a) and aged (b) emulsions showing changes in globules characteristic structures with time: cassava (i), maize (ii) and bentonite (iii) as emulsifying agents at concentration 4%$^{w}/_{v}$ (magnification x 40)

The type of emulsion was identified by the dye stain method using a water-soluble dye (amaranth) and the dispersion was examined microscopically.

Evaluation of the Emulsions.

Determination of globule size and number.
A sample of the dye stained emulsion was diluted 1 in 4 and a few drops smeared on the glass slide mounted on a light microscope (Kyowa, Model: 745917, Tokyo Japan) and examined at a magnification of x40. The number of globule per field of view was counted and sized with the aid of a graduated eyepiece lens from three different fields of view randomly. The mean globule size ($\bar{x}$) was obtained from the expression (Okor 1990 and Eichie *et al.*, 2010) as shown below:

$$\bar{x} = \frac{\Sigma f x}{\Sigma f} \quad \ldots\ldots (1)$$

where f is the frequency of each size x. This procedure was repeated for all types of emulsions after storage for four weeks and the parameters were determined at selected time intervals. Photomicrographs of the emulsions were taken using a digital camera. Triplicate determinations were made in each case and mean values are reported.

In another aspect of the study, The emulsions were also evaluated by storing in calibrated measuring cylinders and stored for a period of six weeks at room temperature 28+2^{0}C followed by periodic examinations in order to determine the rate of creaming as well as phase separation (coalescence), Bullock *et al*., (1992). These parameters were evaluated at selected time intervals for six weeks.

Determination of the viscosity of the emulsions (rheological properties).
Viscosities of the emulsions were determined by the modified method as previously described by Okor and Obaduni 1992 using the Brookfeild DV-E viscometer, in which the viscosities of the emulsions relate directly to the time (s) taken for a sample volume of the emulsion to flow. The viscosity (dynamic) was expressed in centipoises (cP), since the present determination was of comparative value only. The determinations were carried out in triplicate on the various emulsions for six weeks and mean values are recorded.

RESULTS AND DISCUSSION

Rheological properties of mucilages and emulsions. Results of the flow properties of the mucilages and emulsions are shown in Table 1. Generally, the starches displayed higher viscosity values as follows: 1448cP (cassava mucilage), 1140cP (maize mucilage and 36cP (bentonite mucilage) while that of the emulsions were 1730cP (cassava), 1206cP (maize) and 220cP (bentonite) respectively. There was no marked difference in the flow properties of the freshly made mucilages and emulsions stabilized with the starches respectively, although the cassava starch displayed higher viscosities than maize starch. However, this was not the case with bentonite, whose emulsion displayed about eight times the viscosity of its mucilage. For instance, the viscosity of the dispersion medium was 36cP while that of the emulsion was 220cP at same concentration of 4%. There was obvious difference in the flow characteristics exhibited by the starches, compared with bentonite, whose viscosity was about 10 times lower than that of the starches.

Effect of viscosity on globule structure.
Photomicrographs of the emulsion globules revealed that cassava starch formed a more homogenous emulsion characterized with fine sized globules of even distribution (see fig 2a). By comparison maize starch and bentonite mucilages formed coarse emulsions characterized by larger globule sizes and uneven size distribution. The mean globule sizes (µm) per field of view in the emulsions were 12.44 ± 1.2 (cassava), 18.92 ± 2.13 (maize) and 26.17 ± 1.74 (bentonite) respectively.

The probable difference in globule characteristics (globule size and number) of the emulsions is inversely related to differences in the apparent viscocities of the dispersion media. It is thought that on emulsification, the starches and bentonite mucilages gelled and imparted on the viscosity and thus retarded the rate of coalescence of the dispersed globules, and hence the oil globules remain homogenously distributed. Cassava starch with the highest viscosity formed the most stable emulsion, followed by maize and bentonite, which formed coarse, emulsions. Starches generally are regarded as viscosity imparting agents, which act to retard the rate of sedimentation of the dispersed globules and hence stabilize the emulsions. All three stabilizing agents employed imparted immensely on the viscosity of the dispersion medium.

Effect of ageing on the viscosity of the medium. Again photomicrographs of the aged emulsions are shown in Fig 2(b). Upon storage of the emulsions for two weeks, there was a gradual increase in the globule sizes, which declined over the period of storage. It is thought that dispersed globule coalesced to form larger globules which eventually ruptured and disappeared as a result of breakdown of the structured dispersion medium with a resultant loss of emulsification. This effect accounted for the marked reduction in viscosity and hence the rheological properties observed.

Conclusion.

The study has shown that the viscosity of the dispersion medium in emulsified system plays vital role in determining the stability of the resulting emulsions. This finding reveals that the viscosity of the aqueous dispersion medium can be optimized to obtain desirable globule size distributions and hence enhance the stability of the emulsions.

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